FULL PAPER

Multi-wall carbon nanotube supported Co (II) Schiff base complex: an efficient and highly reusable catalyst for synthesis of 1-amidoalkyl-2-naphthol and tetrahydrobenzo[*b*]pyran derivatives

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Sadegh Salehzadeh, Faculty of Chemistry, Bu-Ali Sina University, Hamedan 6517838683, Iran. Email: saleh@basu.ac.ir; ssalehzadeh@gmail.com An immobilized Co (II) Schiff base complex supported on multi-wall carbon nanotubes was synthesized and characterized using Fourier transform infrared spectroscopy, X-ray diffraction, scanning electron microscopy, energy-dispersive X-ray spectroscopy, thermogravimetric analysis and inductively coupled plasma mass spectrometry. It was shown that the supported complex is a facile, eco-friendly, recyclable, reusable and green catalyst for three-component condensation of 2-naphthol and acetamide with various aldehydes for the synthesis of 1-amidoalkyl-2-naphthol derivatives under solvent-free conditions. Also, in a further study, the catalytic application was studied in the synthesis of tetrahydrobenzo[*b*] pyran derivatives via the condensation reaction of malononitrile and dimedone with several aromatic aldehydes. The procedures suggested here for the synthesis of 1-amidoalkyl-2-naphthol and tetrahydrobenzo[*b*]pyran derivatives offer several advantages, such as stability, recyclability and eco-friendliness of the catalyst, simple experimental conditions, short reaction times, high to excellent yields and easy work-up.

KEYWORDS

immobilized Co (II) Schiff base complex supported on MWCNTs, 1-amidoalkyl-2-naphthol derivatives, tetrahydrobenzo[*b*]pyrans, solvent-free

1 | **INTRODUCTION**

Many soluble Schiff base complexes show high valuable catalytic activity in chemical reactions,^[1–3] and also these compounds are efficient catalysts in both homogeneous and heterogeneous systems. Over the past few years, there have been many reports of their applications in homogeneous and heterogeneous catalysis.^[4–8] Immobilization of transition metal complexes on solid supports can be used to prepare catalysts which are easy to be separated from, and reused in, a reaction system and which may possibly exhibit improved activities because of the support environment. Various supports have been reported for the synthesis of a variety of heterogeneous catalysts.^[9,10] Due to good stabilities in both acid and alkaline conditions,

carbon nanotubes as supports have attracted much consideration. $\ensuremath{^{[7]}}$

Multicomponent reactions (MCRs) represent an economic way in one-pot systems to synthesize highly functionalized and complex molecules in a single synthetic operation from simple and readily available starting materials. Thus, MCRs offer high selectivity, high yield and synthetic simplicity due to the formation of carbon–carbon and carbon–heteroatom bonds in a single step.^[11–13] Our target compounds from MCRs were amidoalkylnaphthols, which can be prepared by multicomponent condensation of aldehydes, 2-naphthols and acetonitrile or different amides in the presence of various catalysts.^[14–20] Several heterogeneous catalyst have been reported to catalyse this transformation, such as silica gelsupported $-SO_3H$ -functionalized benzimidazolium,^[21]

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cation-exchanged resins,^[22] silica-supported perchloric acid,^[23] heteropolyacids,^[24] silica gel-supported dual acidic ionic liquid,^[25] sulfanilic acid@MNPs,^[26] FeCl₃@SiO₂,^[27] alumina sulfuric acid^[28] and K₅CoW₁₂O₄₀·3H₂O.^[29] 1-Amidoalkyl-2-naphthol derivatives are important intermediates as an important class of compounds which can be easily converted to biologically and pharmacologically active derivatives by an amide hydrolysis reaction. Also, the depressor and bradycardia effects of these compounds have been evaluated.^[30,31] MCRs are also suitable for the preparation of 4*H*benzo[*b*]pyrans through three-component condensations including the use of SO₄²⁻@MCM-41,^[32] KF-alumina^[33] and Fe₃O₄@MWCNTs^[34] as heterogeneous catalysts.

To the best of our knowledge, the synthesis of 1amidoalkyl-2-naphthols in the presence of transition metal Schiff base complexes has not yet been reported. Herein we report the synthesis and characterization of a Co (II) Schiff base complex immobilized on the surface of multi-wall carbon nanotubes (MWCNTs) through a silicon linker. We report the efficient, rapid and green synthesis of 1amidoalkyl-2-naphthols in the presence of catalytic amounts of this catalyst under solvent-free conditions (Scheme 1). In addition, the synthesized catalyst was efficaciously engaged in the synthesis of numerous tetrahydrobenzo[*b*]pyran derivatives via the condensation reaction of dimedone and malononitrile with several aromatic aldehydes, under solvent-free condition (Scheme 2).

2 | RESULTS AND DISCUSSION

Scheme 3 summarizes the reactions that lead to the Co.(II) Schiff base complex supported on MWCNT nanomaterials. This synthesis was carried out as detailed in the experimental section.

2.1 | Characterization of Immobilized Cobalt(II) Schiff Base Complex Supported on MWCNTs as Heterogeneous Catalyst

The structure of the immobilized cobalt(II) Schiff base complex supported on MWCNTs as a heterogeneous catalyst was



SCHEME 1 Synthesis of 1-amidoalkyl-2-naphthol derivatives using immobilized Co (II) Schiff base complex supported on MWCNTs



SCHEME 2 Synthesis of tetrahydrobenzo[*b*]pyran derivatives using immobilized Co (II) Schiff base complex supported on MWCNTs



SCHEME 3 Sequence of events in the preparation of MWCNTs@Schiff base and its Co (II) complex

studied, considered and identified using Fourier transform infrared (FT-IR) spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive Xray spectroscopy (EDX), and inductively coupled plasma mass spectrometry (ICP).

In order to confirm the surface modification of MWCNTs, the FT-IR spectra of the prepared materials were

obtained and are shown in Figure 1. The FT-IR spectrum of MWCNTs@COOH (Figure 1(a)) shows a strong band at 1710 cm⁻¹ and a weaker band at 1228 cm⁻¹ attributed to C=O and C-OH groups in oxidized MWCNTs, respectively, as well as a broad band at about $3420-3480 \text{ cm}^{-1}$ assigned to O-H stretching vibrations which are not observed in the spectrum of pristine MWCNTs. Comparison studies of MWCNTs@COOH and MWCNTs@APTMS reveal an additional strong band at 1000-1100 cm⁻¹ corresponding to characteristic absorption of Si-O bonds formed through the silvlation process. The presence of anchored propyl chain was confirmed by C-H stretching vibrations appearing at about 2923 cm^{-1} . The presence of these bands suggests that during the anchoring of APTMS, a condensation reaction occurs between the OH surface groups of the oxidized MWCNTs and methoxy groups of APTMS to form the stable covalent Si-O-C linkage, leading to the attachment of aminopropyl groups on the surface of MWCNTs. Also, the band in the spectral region of 1629 cm^{-1} can be assigned to the azomethine peak of the attached salicylaldehyde on the MWCNTs modified with APTMS. In the FT-IR spectrum of the MWCNTs@Co (II) Schiff base, a considerable shift to lower energy (1616 cm⁻¹) is observed in the strong sharp C=N stretching frequency upon complexation with cobalt which suggests the imine nitrogen atoms are coordinated (Figure. 1(d)).

Figure 2 displays the XRD patterns of parent MWCNTs, MWCNTs@Schiff base and MWCNTs@Co (II) Schiff base complex. The XRD patterns of MWCNTs bearing cobalt(II) complex and Schiff base are somewhat similar to that of MWCNTs. This indicates that the crystallinity and morphology of MWCNTs are preserved during the grafting process. Figure 2 shows the XRD pattern of MWCNTs, which is similar to that of highly oriented pyrolytic graphite.^[35] The MWCNTs show typical peaks of (002), (110) and (400) at $2\theta = 26^{\circ}$, 43° and 53°, respectively.



FIGURE 1 FT-IR spectra: (a) MWCNTs@COOH; (b) MWCNTs@APTMS; (c) MWCNTs@Schiff base; (d) MWCNTs@Co (II) Schiff base complex



FIGURE 2 XRD patterns: (a) MWCNTs; (b) MWCNTs@Schiff base; (c) MWCNTs@Co (II) Schiff base complex



FIGURE 3 Field-emission SEM image of MWCNTs@Co (II) Schiff base complex



FIGURE 4 EDX diagram of MWCNTs@Co (II) Schiff base complex



FIGURE 5 TGA curve of MWCNTs@Co (II) Schiff base complex

The structure and morphology of the nanomaterial was analysed using field-emission SEM. The SEM image of immobilized complex supported on MWCNTs is shown in Figure 3. From the micrograph it can be concluded that the nanotubes are aggregated and have retained their nanotube nature. This indicates that the sonication condition used in current study is important and the integrity of the tubes is not destroyed during the treatment. The smooth surface of nanotubes has been altered to a rough surface after the immobilization process, which can be associated with the complex moieties attached to the nanotube surfaces. The functionalization process of the MWCNTs with the Schiff-base and the formation of its complex were also confirmed from the EDX pattern of the product. The presence of Co atoms in the Co.(II) Schiff base complex supported on MWCNTs as nanocatalyst is confirmed by the EDX results which also show the presence of C, O, N, Cl and Si atoms in Figure 4. The Co. content of the immobilized complex supported on MWCNTs was also measured by ICP, the value of which is about 0.623 mmol g⁻¹.

From the thermogravimetric analysis (TGA) curve of Co (II) Schiff base complex supported on MWCNTs (Figure 5), a total weight loss of *ca* 20% is observed on heating to 700 ° C. These studies have been made in the temperature range 50–1000 °C in nitrogen atmosphere. Degassing and solvent evaporation at low temperatures account for approximately 1% of the loss. The majority of the weight loss occurs at temperatures >450 °C. However, for the corresponding supported complex, 3 and 8% weight losses appear at temperatures of 209 and 368 °C, respectively.

2.2 | Application of Immobilized Co (II) Schiff base Complex Supported on MWCNTs as Heterogeneous Catalyst

At first, screening trials were performed to optimize various reaction parameters, including catalyst amount, solvent and

	H_3C O + H_2N	H Amount of nano Cat (?) Solvent-free, Temperature (?)	NH OCH3	
Entry	Amount of Catalyst (g)	Reaction temperature (°C)	Reaction time (min)	Yield (%) ^b
1	—	r.t.	120	—
2	—	100	120	—
3	0.001	r.t.	120	5
4	0.001	100	120	38
5	0.002	r.t.	120	7
6	0.002	100	120	51
7	0.005	r.t.	60	10
8	0.005	100	60	65
9	0.01	r.t.	60	15
10	0.01	50	60	63
11	0.01	75	25	93
12	0.01	100	25	93
13	0.01	125	25	93
14	0.02	r.t.	60	15
15	0.02	100	25	93

TABLE 1 Effect of amount of catalyst and temperature on condensation reaction of biphenyl-4-carbaldehyde, β -naphthol and acetamide under solvent-free conditions^a

 a Reaction conditions: biphenyl-4-carbaldehyde (1 mmol), β -naphthol (1 mmol), acetamide (1.2 mmol).

^bIsolated yield.

TABLE 2	Effect of solvent on reaction of biphenyl-4-carbaldehyde, f	}-
naphthol a	d acetamide ^a	

H ₃ C H ₂ N	0 + H	Nano Cat (0.01 g) Solvent (?), 75 °C	NH OCH3
Entry	Solvent	Reaction time (min)	Yield (%) ^b
1	Solvent-free	25	93
2	H_2O	120	
3	C ₂ H ₅ OH	60	31
4	CH ₃ CN	45	57
5	CH_2Cl_2	45	60
6	Toluene	120	—
7	CH ₃ CO ₂ Et	120	18
8	<i>n</i> -Hexane	120	

^aReaction conditions: biphenyl-4-carbaldehyde (1 mmol), β -naphthol (1 mmol), acetamide (1.2 mmol), 75 °C, catalyst (0.01 g).

^bIsolated yield.

temperature, with the results being summarized in Tables 1 and 2. After the synthesis, characterization and identification of the heterogeneous catalyst, we considered the efficacy of -WILEY-Organometallic 5 Chemistry

the catalyst in the synthesis of amidoalkylnaphthol derivatives. Thus the solvent-free condensation of biphenyl-4carbaldehyde, β -naphthol and acetamide (model reaction) was examined in the presence of catalyst in the temperature range 25-120 °C (Table 1). We also examined the influence of catalyst loading (Table 1). The model condensation reaction was tested in the presence of various amounts of Co (II) complex supported on MWCNTs under similar conditions. For the optimized amount of catalyst, we found that 0.01 g of nanocatalyst effectively catalyses the reaction for the synthesis of the desired product. The same reaction without nanocatalyst does not lead to product (Table 1, entries 1 and 2). Thus the catalytic efficiency of this catalyst was definitely identified. In view of environmentally friendly methodologies, we have discovered that this reaction proceeds very efficiently by stirring a mixture of neat reactants at 75 °C for only 25 min, under solvent-free conditions, and produces amidoalkylnaphthol derivatives in high yields (Table 1, entry 11).

In the next step, in order to check the effect of solvents in the model reaction, various solvents were used. As evident from Table 2, the reaction proceeds most readily to give the highest yield of the desired product under solvent-free conditions.

TABLE 3 Three-component synthesis of amidoalkylnaphthol derivatives using 0.01 g of nanocatalyst^a

		O O H + O R': CH ₃ , NH ₂ , Ph	Nano Cat (0.01 g) Solvent-free, 75 °C	R H O R'	
Entry	R	R'	Time (min)	Yield (%) ^b	M.p. (°C) (colour)
1	4-NO ₂	CH ₃	20	95	245-247 (yellow) ^[36]
2	4-NO ₂	—Ph	25	93	254-256 (white) ^[36]
3	4-NO ₂	$-NH_2$	30	90	201-203 (yellow) ^[36]
4	4-C1	CH ₃	25	93	261-263 (white) ^[36]
5	4-C1	—Ph	30	91	208-210 (pink) ^[36]
6	4-C1	$-NH_2$	35	88	163-165 (white) ^[36]
7	2,5-Dimethoxy	CH ₃	30	90	283-285 (white) ^[36]
8	2,5-Dimethoxy	—Ph	35	89	274-276 (pink) ^[39]
9	2,5-Dimethoxy	$-NH_2$	40	87	209-211 (yellow) ^[39]
10	1-Naphthyl	CH ₃	25	92	263-265 (white) ^[39]
11	1-Naphthyl	—Ph	30	90	291-293 (white) ^[39]
12	1-Naphthyl	$-NH_2$	35	88	199-201 (cream) ^[36]
13	2-Naphthyl	CH ₃	25	92	253-255 (pink) ^[37]
14	2-Naphthyl	—Ph	30	90	266-268 (pink) ^[39]
15	2-Furyl	$-NH_2$	30	89	140-142 (white) ^[36]
16	2-Thienyl	$-NH_2$	30	89	142-154 (white) ^[36]
17	4-Cinnamyl	CH ₃	20	90	230–232 (yellow) ^[38]
18	4-Cinnamyl	—Ph	20	90	201–203 (yellow) ^[39]
19	4-Ph	CH ₃	25	93	245-247 (white) ^[39]
20	4-Ph	—Ph	30	91	285-287 (white) ^[39]

^aReaction conditions: aldehyde (1 mmol), β-naphthol (1 mmol), amide derivatives (1.2 mmol), 75 °C, solvent-free.

^bIsolated yield.

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After optimization of the reaction conditions, we studied the generality of these conditions with a series of aromatic aldehydes under the optimized conditions (Table 1, entry 11; Table 2, entry 1), and the results are summarized in Table 3. We extended our study using MWCNTs@Co (II) Schiff base complex (0.01 g) in the absence of solvent with various aromatic aldehydes to prepare a series of 1-amidoalkyl-2-naphthol derivatives. A variety of differently substituted aromatic aldehydes possessing electron-donating and electron-withdrawing groups give good yields (87-95%) and the reactions are completed within 20-40 min under solvent-free conditions. Several aromatic aldehydes containing electron-releasing substituents, electron-withdrawing substituents and halogens on their aromatic ring were utilized in the reaction, and give the corresponding products in high yields and in short reaction times. As evident from Table 3, the ease of the reaction is directly related to the substituents attached to the benzene ring. The electron-withdrawing groups (such as nitro and halide) are found to activate the aldehyde towards nucleophilic attack and increase the reaction rate (entries 1-6).

Scheme 4 shows a possible mechanism^[40] for the synthesis of 1-amidoalkyl-2-naphthol derivatives in the presence of MWCNTs@Co (II) Schiff base complex as catalyst. The mechanism begins with the activation of the carbonyl group of the aldehyde with the immobilized catalyst and ends with the reaction of *o*-quinone methides (*o*-QMs) with amides or urea to form the 1-amidoalkyl-2-naphthol derivatives.

In another study, in order to establish the general application of our catalyst, we extended the scope of the reaction to the synthesis of various tetrahydrobenzo[b]pyran derivatives. We used the above-mentioned optimized condition to evaluate the condensation reaction of malononitrile and dimedone with a series of aromatic aldehydes under solvent-free conditions.



SCHEME 4 Proposed mechanism for the synthesis of 1-amidoalkyl-2naphthol using immobilized cobalt(II) Schiff base complex supported on MWCNTs as a heterogeneous catalyst

The results are summarized in Table 4. Tetrahydrobenzo[*b*] pyran derivatives containing electron-withdrawing groups such as nitro and halide groups or electron-donating groups such as hydroxyl and alkoxy groups are formed in a short reaction time (15–35 min) with excellent yields (90–95%).

The reusability of the catalyst was investigated in the condensation of biphenyl-4-carbaldehyde, β -naphthol and acetamide. At the end of the reaction, ethyl acetate was added to the reaction mixture and heated to extract the product from the catalyst (the product is soluble in hot ethyl acetate and the catalyst is insoluble). As shown in Figure 6, the recovered catalyst was reused for seven successive runs without significant loss of its catalytic activity. The general methods to determine the presence or absence of leached soluble cobalt in the reaction solution are a hot filtration test or elemental analysis of the recovered catalyst. The heterogeneity of the

TABLE 4 Three-component synthesis of tetrahydrobenzo[b]pyran derivatives using 0.01 g of nanocatalyst^a

	CHO R R	Nano catalyst (0.01 g) Solvent-free, 75 °C	R NH ₂	
Entry	R	Time (min)	Yield (%) ^b	M.p. (°C) (colour)
1	4-NO ₂	15	95	164-166 (white) ^[32]
2	4-Cl	20	93	231-233 (yellow) ^[32]
3	1-Naphthyl	20	93	230-232 (yellow) ^[41]
4	2-Naphthyl	20	93	284-286 (yellow) ^[42]
5	4-Hydroxy	35	91	245-247 (yellow) ^[43]
6	4-Ph	20	93	275–277 (yellow) ^[42]
7	3-NO ₂	20	93	224-226 (white) ^[44]
8	4-Methoxy	30	92	216-218 (white) ^[32]
9	2-Methoxy	30	90	196-198 (white) ^[45]
10	2,4-Dichloro	20	91	221-223(yellow) ^[34]

^aReaction conditions: aldehyde (1 mmol), dimedone (1 mmol), malononitrile (1 mmol), 75 °C, solvent-free.

^bIsolated yield.



FIGURE 6 Study of cobalt(II) Schiff base complex supported on MWCNTs after seven catalytic cycles: (a) SEM image; (b) FT-IR spectrum; (c) EDX pattern; (d) XRD pattern

nanocatalyst in solvent-free condition is obvious and in such condition hot filtration is useful. ICP analysis of the product after isolation of catalyst shows no loss of metal (cobalt) during the catalytic reaction, indicating that no metal leaching occurs. It is also observed from spectral studies (Figure 7) that there is no change in the nature of the catalyst even after seven cycles. The SEM images of catalyst before (Figure 3)



FIGURE 7 Reusability of immobilized cobalt(II) Schiff base complex supported on MWCNTs as a heterogeneous catalyst in 25 min

and after (Figure 7(a)) the reactions show identical shapes. Likewise, the FT-IR and powder XRD analyses exhibit identical peaks for both the fresh and recovered catalyst (Figures 7 (b) and (d)). These experimental results clearly suggest that the reaction involves a heterogeneous process and there is no significant change in the catalytic activity of the nanocatalyst after its catalytic application.

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3 | CONCLUSIONS

Immobilized Co (II) Schiff base complex supported on MWCNTs was synthesized, characterized and used as a highly efficient and green nanocatalyst for the synthesis of 1-amidoalkyl-2-naphthol and tetrahydrobenzo[*b*]pyran derivatives under solvent-free conditions at 75 °C. We have described herein a comprehensive methodology for the synthesis of these derivatives using various structurally different aldehydes in good to excellent isolated yields. Simplicity in process, short reaction time, avoiding the use of any base or Lewis acid catalyst, absence of side products, ease of product isolation and low costs are some of the prominent features of these reactions with our catalyst.

4.1 | Immobilized Co.(II) Schiff base complex on MWCNTs: preparation and purification

4.1.1 | MWCNT functionalization and purification

Following careful purification, MWCNTs were oxidized in a mixture of concentrated sulfuric and nitric acids (3:1, 98 and 70%, respectively) by ultrasonication for 12 h to obtain oxidized MWCNTs (MWCNTs@COOH).^[35] To increase the reactivity and/or population of OH surface groups, the resulting material was further treated with sodium borohydride in methanol and carboxyl groups were reduced to CH₂OH groups.

4.1.2 | Preparation of MWCNTs@NH₂

The as-prepared MWCNTs@OH (1 g) was dispersed in 100 ml of xylene and loaded in a 250 ml round-bottom flask, followed by stirring and ultrasonication (25 W, 40 kHz) for 1 h. Subsequently, APTMS (excess) was added dropwise into the suspension. The mixture was stirred and refluxed for 24 h under dry nitrogen atmosphere. After cooling to room temperature, the prepared MWCNTs@NH₂ was filtered and washed with absolute ethanol and water three times to remove the unreacted residue of silylating reagent and then dried at 80 °C.

4.1.3 | Preparation of MWCNTs@Schiff base and its Co.(II) complex

The as-prepared MWCNTs@NH₂ was dispersed in 60 ml of absolute ethanol. Excess salicylaldehyde and a few drops of acetic acid as a catalyst were added into the suspension. After refluxing for 12 h, a black solid was obtained on filtering the mixture and washing with methanol twice to remove the unreacted residue of salicylaldehyde. Finally, the solid was mixed with Co(OAC)₂ in methanol containing a few drops of triethylamine and refluxed for 24 h at room temperature. The resulting mixture was filtered and washed with hot methanol, ethanol, and water three times, successively, until solutions became colourless. The obtained product was dried and stored in a vacuum desiccator (Scheme 3).

4.2 | General Procedure for Synthesis of Amidoalkylnaphthol Derivatives

To a mixture of aromatic aldehyde (1 mmol), β -naphthol (1 mmol) and amide derivative (1 mmol) in a round-bottom flask was added the immobilized Co (II) Schiff base complex supported on MWCNTs as a heterogeneous catalyst (0.01 g). The resulting mixture was firstly stirred magnetically under solvent-free conditions at 100 °C. After completion of the reaction, as checked via TLC (*n*-hexane–ethyl acetate, 5/2), ethyl acetate (10 ml) was added to the reaction mixture, stirred and refluxed for 3 min, and was then filtered to separate the catalyst from the other materials. The solvent of the organic layer was evaporated and the crude product was

4.3 | General Procedure for Synthesis of Tetrahydrobenzo[*b*]pyran Derivatives

A mixture of aromatic aldehyde (1 mmol), malononitrile (1 mmol), dimedone (1 mmol) and Co (II) Schiff base complex supported on MWCNTs (0.01 g) was stirred under solvent-free conditions for a suitable time at 75 °C. After completion of the reaction as indicated by TLC (*n*-hexane–ethyl acetate, 5/2), ethyl acetate (10 ml) was added to reaction mixture, stirred and refluxed for 3 min, and was then filtered to separate the catalyst from the other materials. The solvent of the organic layer was evaporated and the crude product was purified by recrystallization from ethanol.

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